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ALTERNATING CURRENT (AC) IMPEDANCE TESTING OF COATED TRAYCANS

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13. ABSTRACT (*Maximum 200 words*)

This is the final report on Cortest Columbus Technologies' program entitled "AC Impedance Testing of Coated Traycans", performed under Contract DAAK60-90-1301. The overall objective of the program was to evaluate the relative resistance of several candidate coatings prior to retorting to a solution containing NaCl and citric acid (simulating saline, acidic food product) at ambient temperature. An additional objective of the program was to perform an initial assessment of the applicability of the AC impedance technique as a quality assurance technique for traycan coatings. The performance of the coatings was evaluated by means of an Electrochemical Impedance Spectroscopy (EIS) technique, also referred to as AC impedance.

The EIS technique was found to be a sensitive technique for measuring coating degradation on the traycans. Of the coatings analyzed, the VMC coating, in general was found to be the best performer, followed closely by DMS and VMS. The control coating as found to be the poorest coating, of the coatings analyzed. No measurable effect of forming the corners of traycans on coating performance was found in the study. The EIS technique is promising for quality control but further research is needed to optimize the analysis time and simplify the test technique.

14. SUBJECT TERMS

Traycan	Tinplating	AC Impedance
Corrosion Resistance	Corrosion	Tin-free Steel
Electrochemical Impedance Spectroscopy		Coatings
		Test and Evaluation

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INTRODUCTION

This is the final report on Cortest Columbus Technologies' program entitled "AC Impedance Testing of Coated Traycans", performed under Natick Contract DAAK60-90-1301. The overall objective of the program was to evaluate the relative resistance of several candidate coatings prior to retorting to a solution containing NaCl and citric acid (simulating a saline acidic food product) using the AC impedance technique. An additional objective of the program was to perform an initial assessment of the applicability of the AC impedance technique as a quality assurance technique for the traycan coatings.

PROCEDURE

a. Apparatus. The apparatus used by Cortest Columbus consists of a frequency response analyzer used in conjunction with a potentiostat connected to a microcomputer for data analysis and plotting. For the Natick contract, the test cell consisted of a traycan partitioned into three areas by means of dividers and foam gaskets to represent different configurations of the traycan surface. The test areas were filled with a three per cent solution of NaCl in deionized water adjusted to a pH of 4-5 to simulate a saline, acidic, aggressive food environment such as a tomato paste. The temperature was ambient and conditions were aerobic. The cell was covered with plastic film wrap to control evaporation (losses were made up with deionized water). There were seven traycan cells set up for testing at one time.

b. Coatings Studied. The coatings studied were the four variables considered in the Natick Traycan Improvement Program plus the current traycan. They represented the four candidate coatings applied on 0.75 tinplate traycans and the current traycan made of coated tin-free steel as the control. A detailed description of the coating variables is shown in Table 1. For

Table 1. Coated Traycans Tested

	Designation	Exterior Coat	Base Coat	Interior Coat
1.	Dexter Matte Sheet (DMS)*	Aluminum Vinyl	Epoxy Phenolic	Aluminum Vinyl
2.	Reliance Matte Sheet (RMS)*	Aluminum Epoxy	Clear Epoxy	Aluminum Vinyl
3.	Valspar Matte Sheet (VMS)*	Clear Epoxy	Clear Vinyl	Aluminum Vinyl-High Solids
4.	Valspar Matte Coil (VMC)*	Clear Epoxy	Clear Epoxy	Aluminum Vinyl-High Solids
5.	Valspar over Tin Free Steel (Control-Ctr)	Clear Epoxy	Clear Epoxy	White Vinyl

*Tin Plate Substrate - 90 lb per base box Electrolytic Tin Plate, Matte Finish, 0.75/0.35 tin weights.

identification purposes, candidate coatings were designated as Dexter Midland Matte Sheet (DMS), Reliance Matte Sheet (RMS), Valspar Matte Sheet (VMS) and Valspar Matte Coil (VMC). The control was designated as CTR.

c. Testing sequence was as follows:

(1) The first test run of cells consisted of duplicates of CTR and VMS coatings and one each of DMS, VMC and RMS coatings. After 1200 hours, this run was interrupted to allow setting up two different cells (Run #2, Table 2). CTR-1, VMS-2, and RMS coatings were terminated to provide space for Run #2 coating tests.

(2) The second test run of shorter duration, 500 hours, was conducted on a third control (CTR-3), and an abraded VMS to determine the effect of slight surface mechanical damage. A new control was also tested in this run, but was dropped from the program as the corrosion resistance was inferior.

(3) The remaining four of the original runs, CTR, VMS, DMS, and VMC continued to be tested for a total of 1900 hours. Table 2 outlines the test sequence:

Table 2. Summary of Tests

Run #	Coating	Duration of Test, Hours
1	CTR-1	1200
	CTR-2	"
	VMS-1	"
	VMS-2	"
	RMS	"
	DMS	"
	VMC	"
2	New Control*	500
	VMS-Abraded	500

CTR-3

Run #1 continued

VMS-1	1900	Total
CTR-2	1900	
DMS	1900	
VMC	1900	

* Dropped from program due to poor performance.

d. AC Impedance.

Test Technique.

A series of small AC voltages, less than 20 millivolts, were applied to the coated specimen by means of a platinum counter electrode. Using the potentiostat, the frequency response analyzer analyzed the correspondent lead or log angle (phase shift, similar to power factor) and the AC impedance (similar to DC resistance) at each frequency of applied AC voltage. The computer was fed these data and calculated the impedance or resistance at each frequency and plotted these data for each exposure time being measured. This is called a Bode plot (Figure 1). Polarization or total resistance was obtained from the Bode plot by determining the impedance values for each measurement at the low frequency limit as shown in Figure 1. These data were plotted versus time in Figures 4-12 and for each coating in Figures 13-15 for 500, 1000 and 1500 hour exposure periods.

RESULTS

a. Bode Plots. Figures 1-3 represent the Bode plots after 430 and 1872 hours on two coatings tested. A Bode plot is a graph of the log of Z, the impedance or AC resistance versus the log of the frequency at which each measurement was made. The phase angle was also plotted versus frequency in

Figures 1-3. However, these plots were not used for making the final conclusions. As stated under "Procedure", the total system resistance, the low frequency limit for the polarization resistance, Z, was obtained for each Bode plot representing a specific coating and exposure time. This extrapolation procedure is illustrated in Figure 1.

b. Total Polarization Resistance versus Time Plots. These data are shown in Figures 4-12 and are obtained from Bode plots. These plots show the change in total resistance (corresponding to corrosion resistance) with exposure time for each coating.

c. Total Resistance versus Coating Type after 500, 1000, and 1500 Hours Exposure, Figures 13-15, respectively, illustrate these data after the three time periods. These data were obtained from the plots of total resistance versus time.

DISCUSSION

The objective of the investigation was to measure the overall performance of each coating by means of measuring the overall corrosion resistance versus time using the AC Impedance technique. In order to simplify the program, details obtained using this technique such as the Nyquist plots and phase angle versus frequency, which are useful in analyzing sub-components of the total resistance, are not included or considered herein. These subcomponents are solution, substrate, and pore resistance. Since the total resistance of the system to corrosion is the primary quantity of interest, only this information was used to reach the conclusions.

The results that lead to the following conclusions were the total resistance versus time plots (Figures 4-12) and the comparison of the resistance of the coatings tested after various exposures times (Figure 13-15). These data show that the VMC coating consistently exhibited the best preretort corrosion resistance of the coatings evaluated, exhibiting high resistance values throughout the testing. A fall in resistance versus time is indicative of coating degradation. VMC exhibited only a slight decrease in resistance after 1500 hours of exposure. The DMS and VMS coating also performed well in the testing. The resistance of the DMS coating after 500 hours was comparable to that of the VMC coating, but the DMS coating degraded somewhat faster than the VMC coating thereafter. The VMS coating exhibited somewhat lower resistances than the VMC or DMS coatings and slowly degraded over the testing period. The remaining coatings, RMS and Control, exhibited lower performance than any of the tested coatings with Control showing the most rapid degradation. As anticipated, the abraded VMS coating exhibited very low resistances, demonstrating the expected values for a completely failed coating. For any of the coatings, comparison of the data for the three compartments indicates that there was no measurable effect of formed corners on coating performance.

VMC was considered by Central States Can Co., Massillon, Ohio to be the best of the coating candidates except for poor adhesion at formed corners of the traycan body. The next best, DMS, did not exhibit poor adhesion at the formed traycan corners.

CONCLUSIONS

(1) The AC Impedance technique was found to be a sensitive technique for measuring coating degradation on traycans.

(2) Of the coatings analyzed, the VMC coating was found to be the best performer, followed closely by the DMS and the VMS coatings. These conclusions approximated those reached in the Ross report[1].

(3) The Control coating was found to be the poorest coating, of the coatings analyzed.

(4) No measurable effect of forming the corners of the traycans on coating performance was found in the study. As mentioned above, VMC was reported to have poorer adhesion at the formed corners of the traycan body when compared to DMS.

(5) The AC Impedance technique is promising for quality control but further research is needed to optimize the analysis time and simplify the test technique.

It should be cautioned that the preceding conclusions are based on the long-term ambient temperature exposures and do not consider blistering or coating degradation associated with the high temperature thermal process to which filled and sealed traycans are subjected.

This document reports research undertaken at the US Army Natick Research, Development and Engineering Center and has been assigned No. NATICK/TR-95/019 in the series of reports approved for publication.

REFERENCE

1. Ross, Jeanne M. Evaluation of Half Steam Tray, Tinplate vs Tin-free steel, Phase I, Technical Report TR-91/030 U.S. Army Natick RD&E Center, FED, Nov 90

APPENDIX

Figures

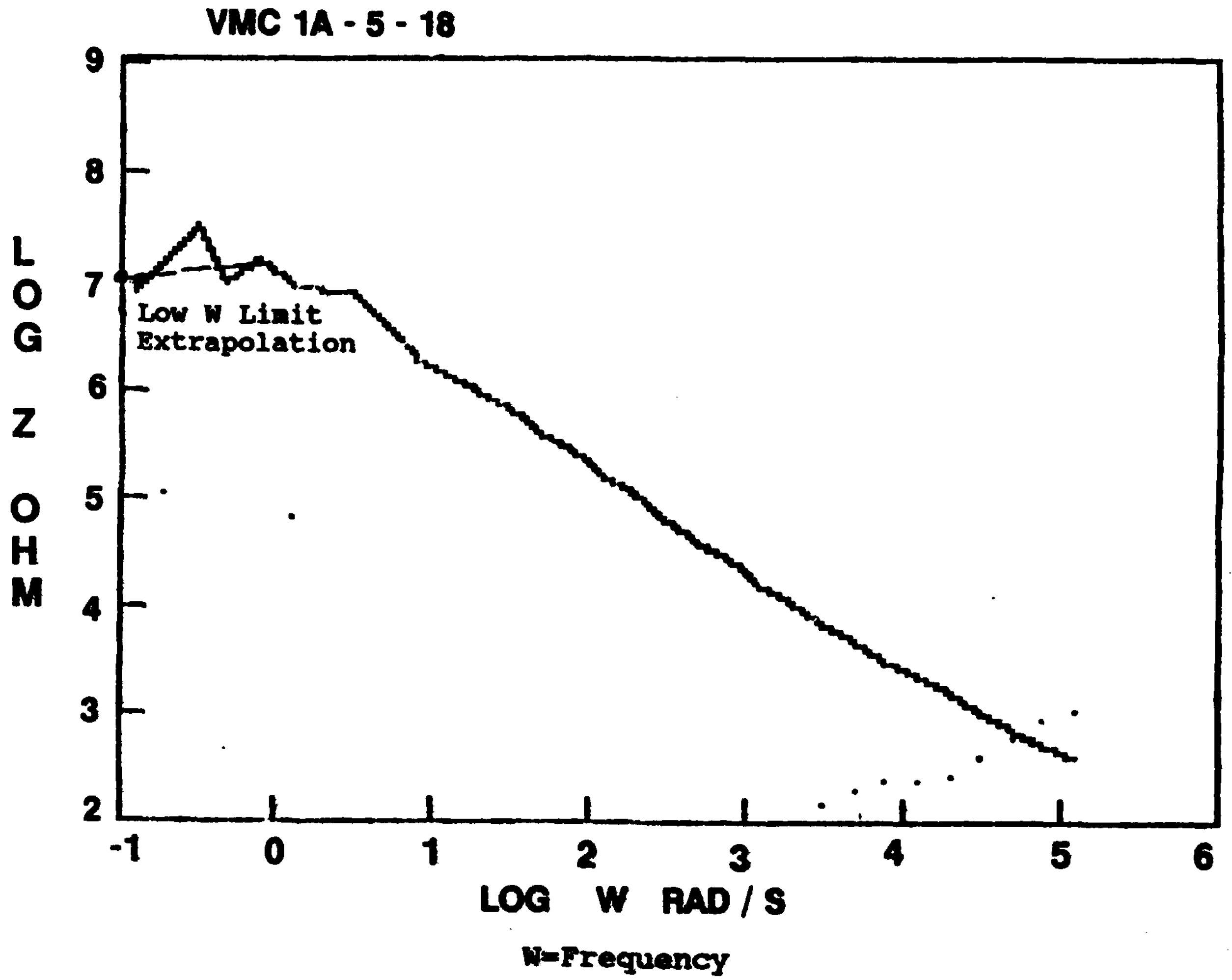


Fig. A-1. Bode Plot for VMC Coating After 430 Hours of Exposure

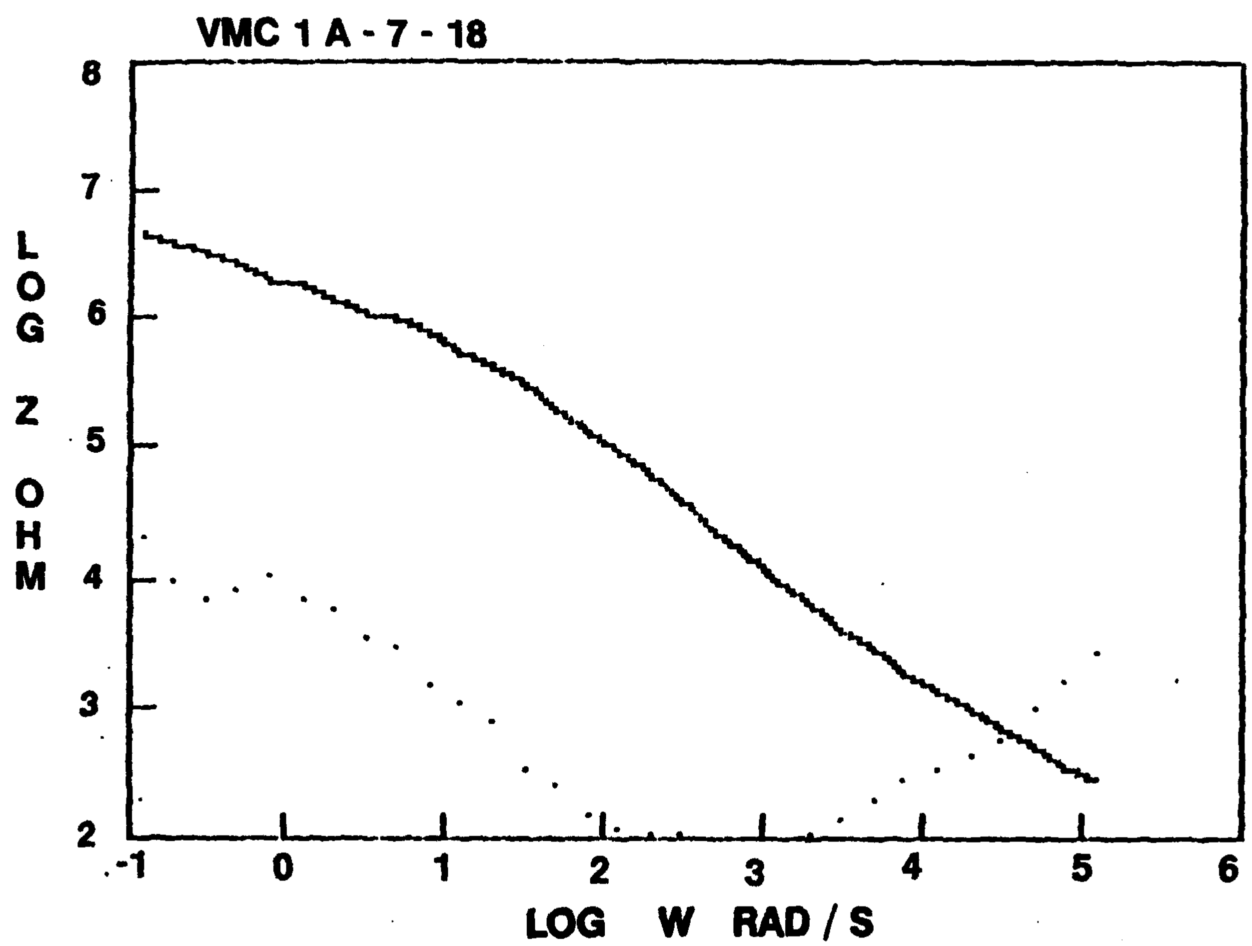


Fig. A-2. Bode Plot for VMC Coating After 1872 Hours of Exposure

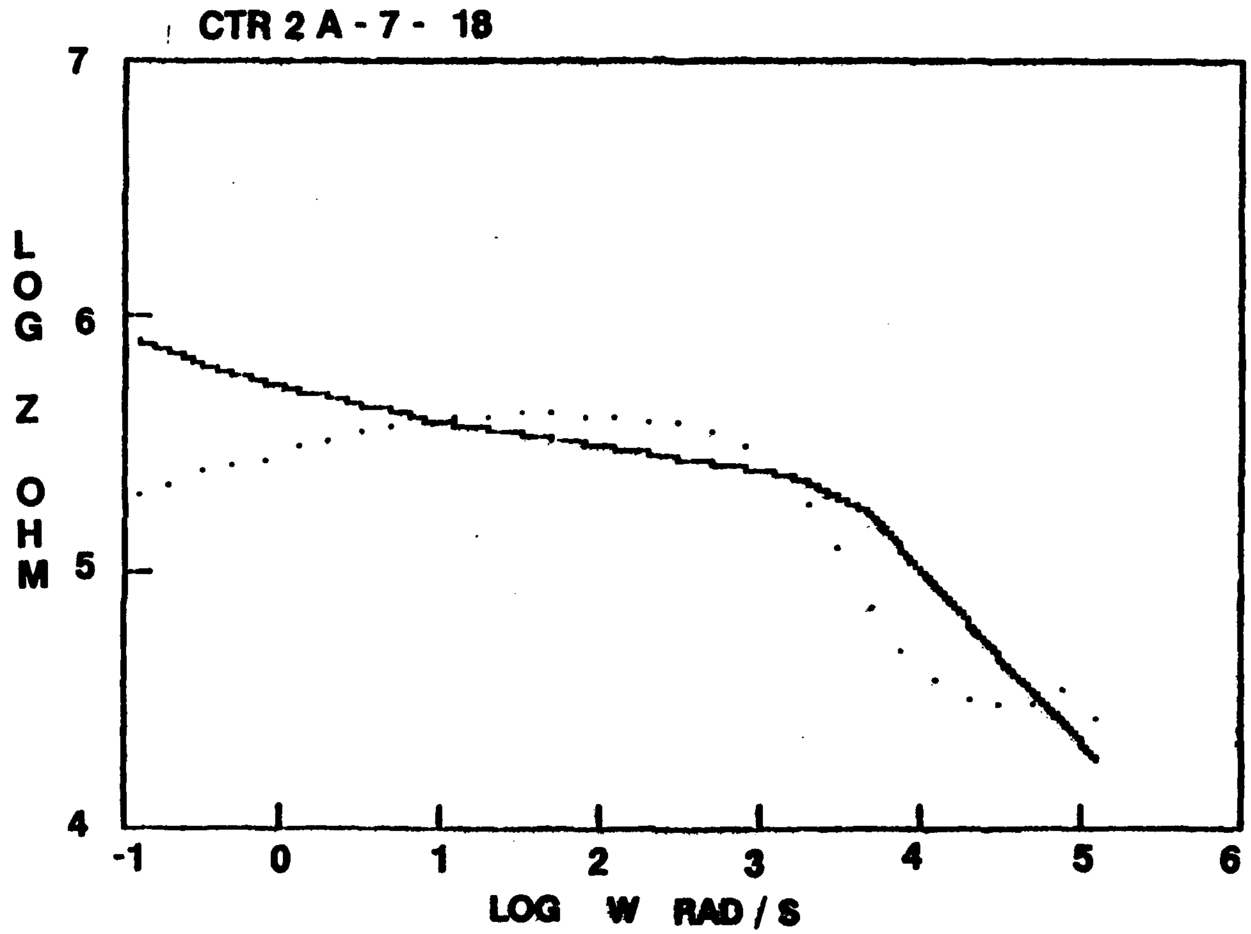


Fig. A-3. Bode Plot for Control Coating After 1872 Hours of Exposure

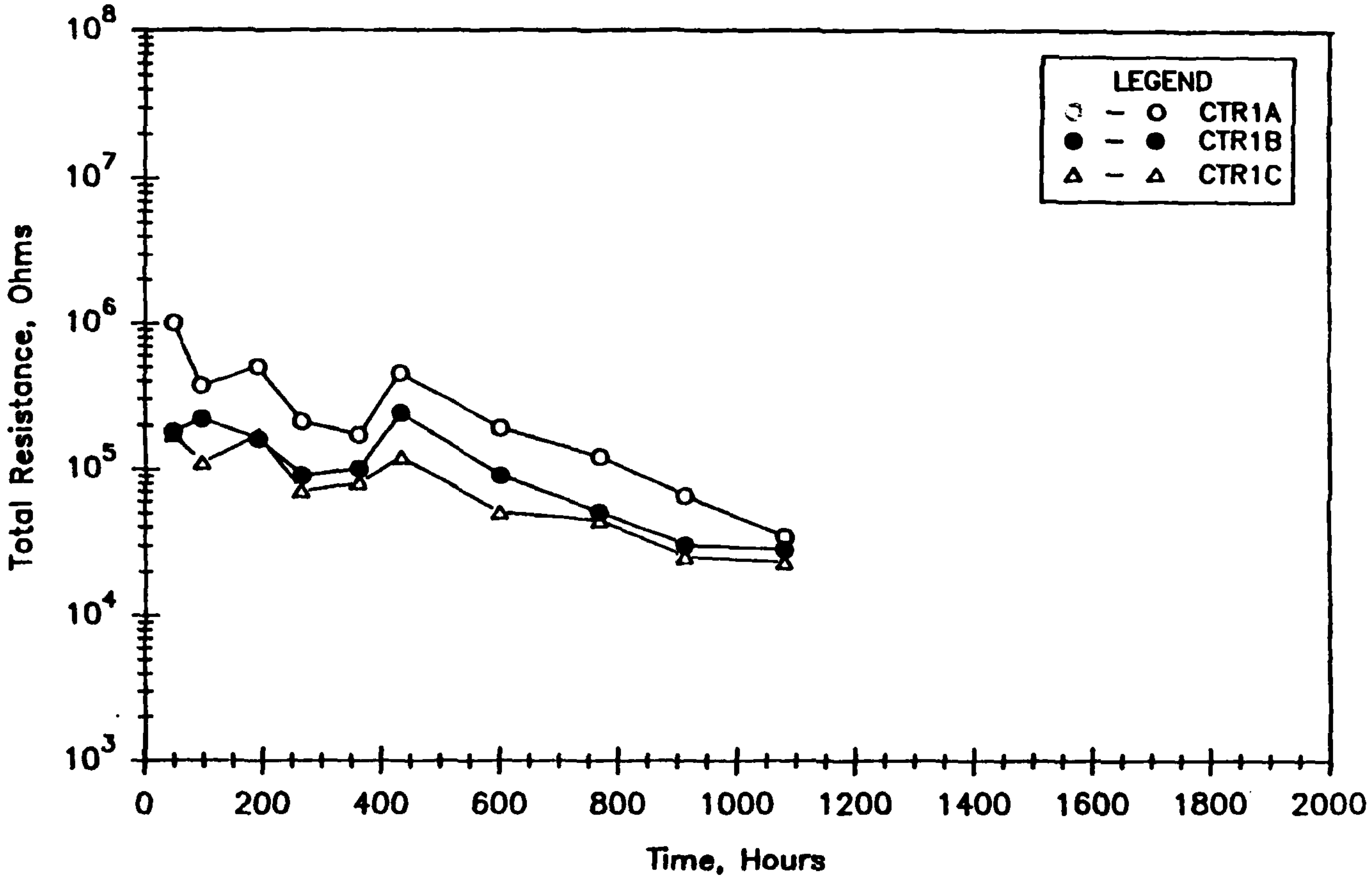


Fig. A-4. Total Resistance as a Function of Time for Control Coating (CTR 1). A and C were Corner Compartments, B was the Center Compartment.

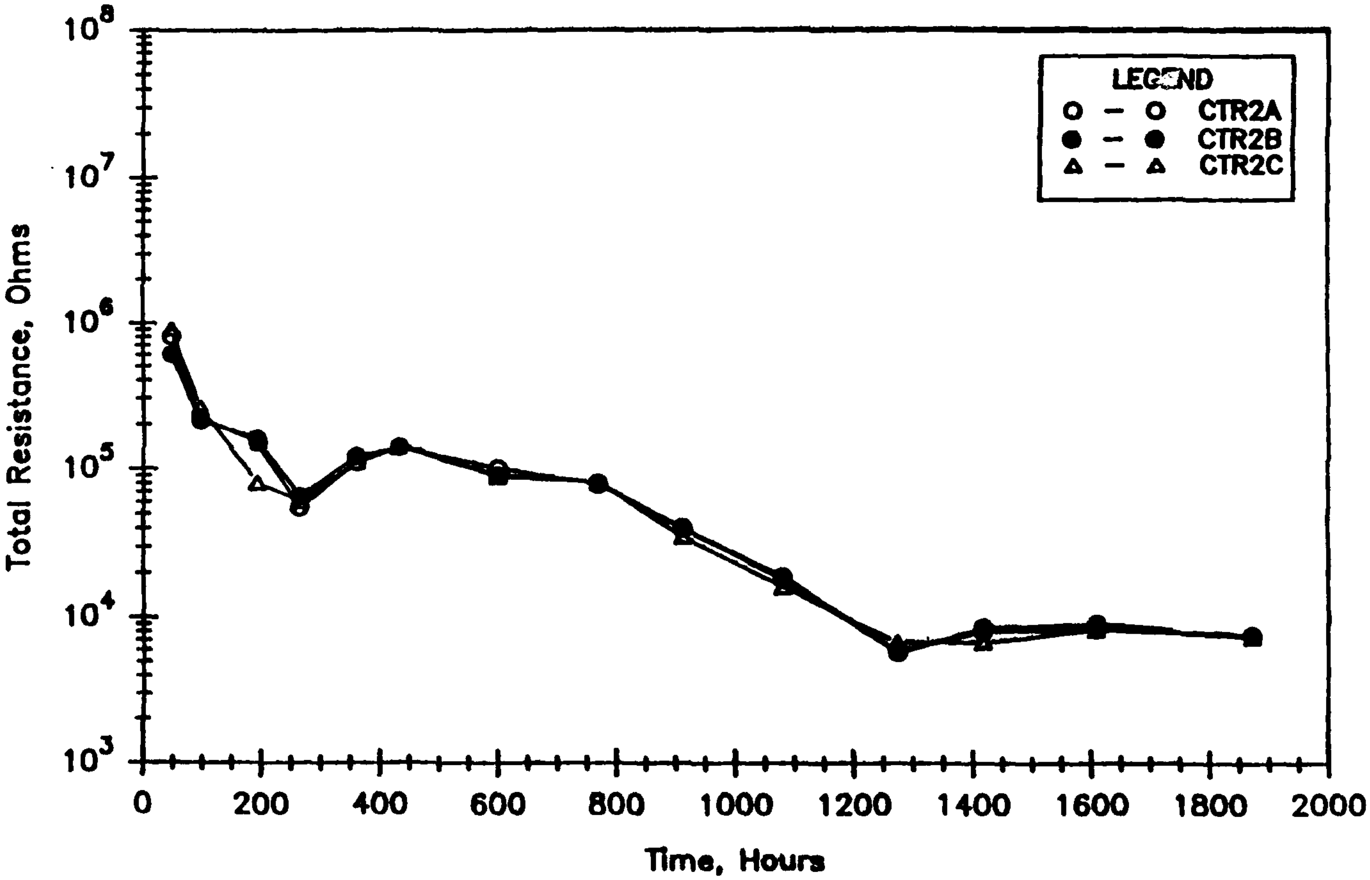


Fig. A-5. Total Resistance as a Function of Time for Control Coating (CTR 2). A and C were Corner Compartments, B was the Center Compartment.

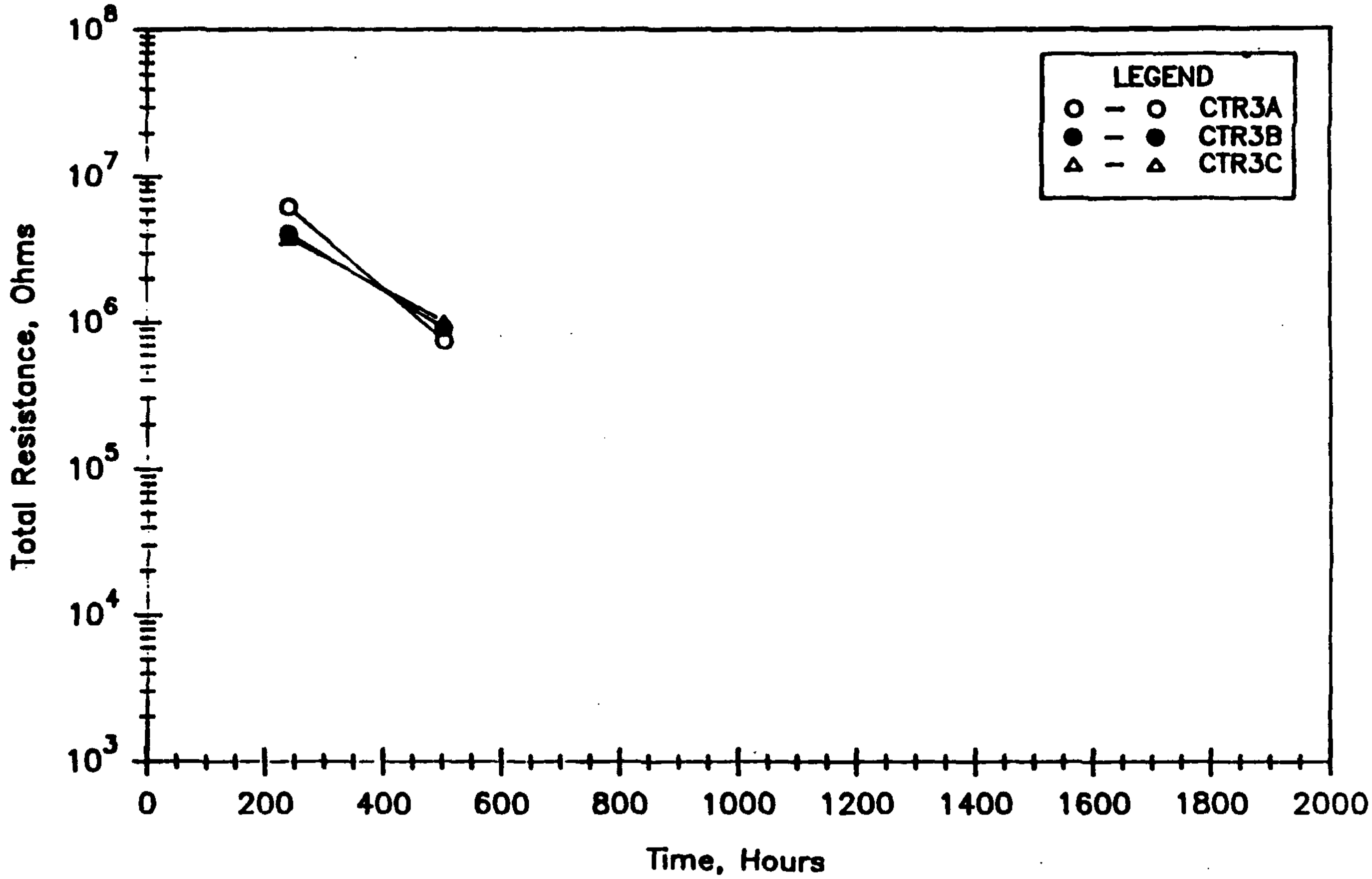


Fig. A-6. Total Resistance as a Function of Time for Control Coating (CTR 3). A and C were Corner Compartments, B was the Center Compartment.

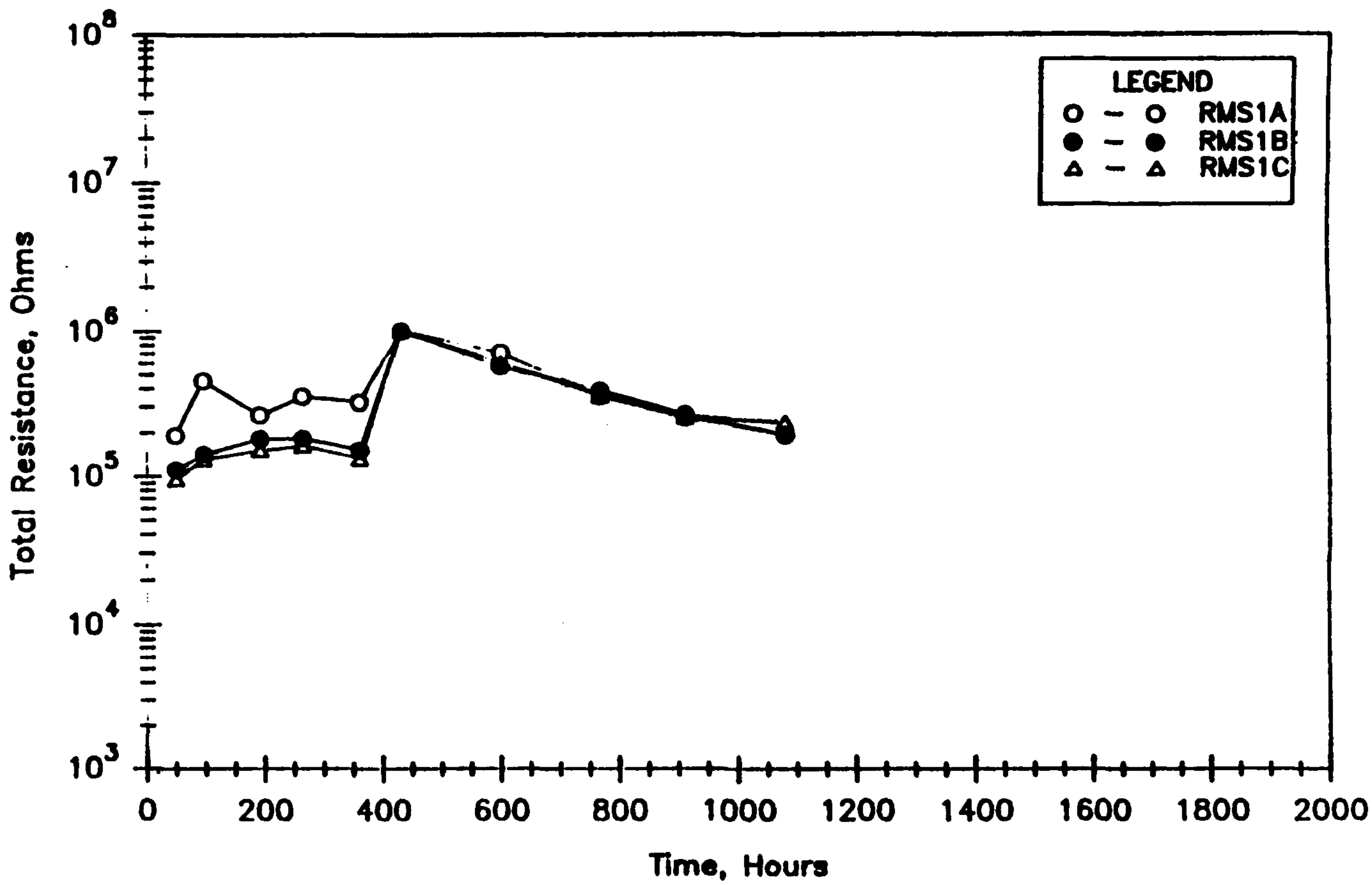


Fig. A-7. Total Resistance as a Function of Time for RMS Coating 1. A and C were Corner Compartments, B was the Center Compartment.

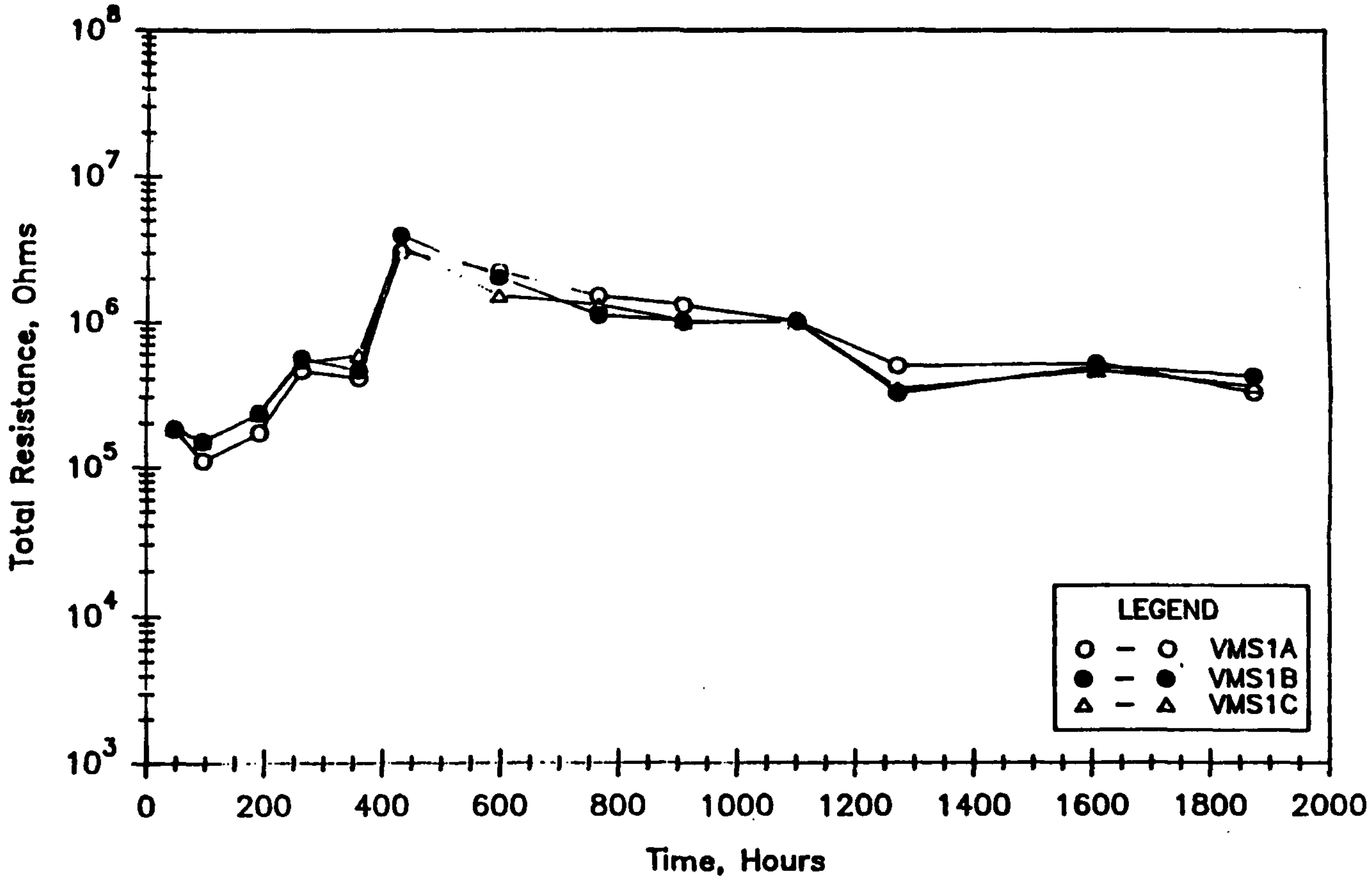


Fig. A-8. Total Resistance as a Function of Time for VMS Coating 1. A and C were Corner Compartments, B was the Center Compartment.

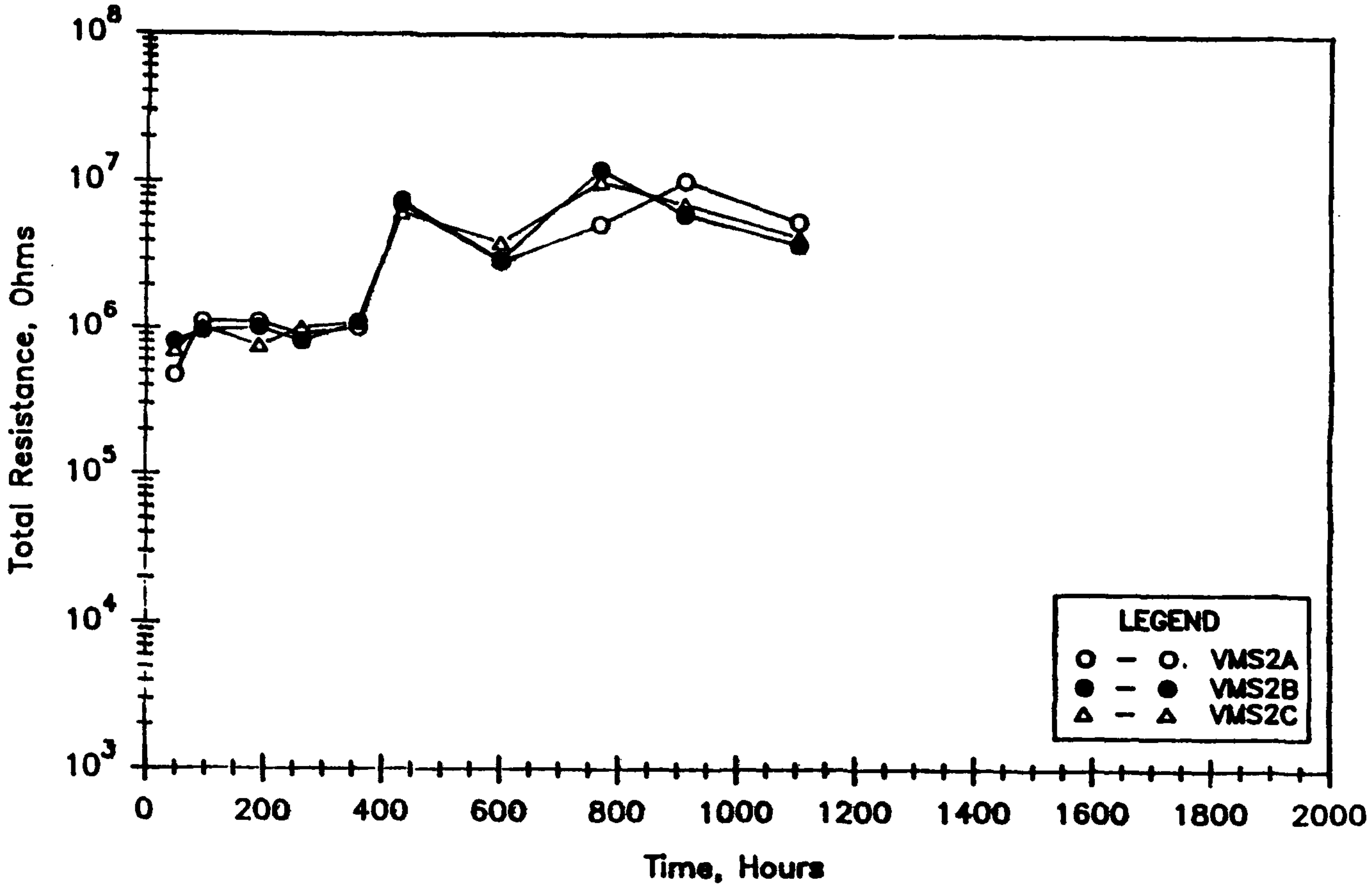
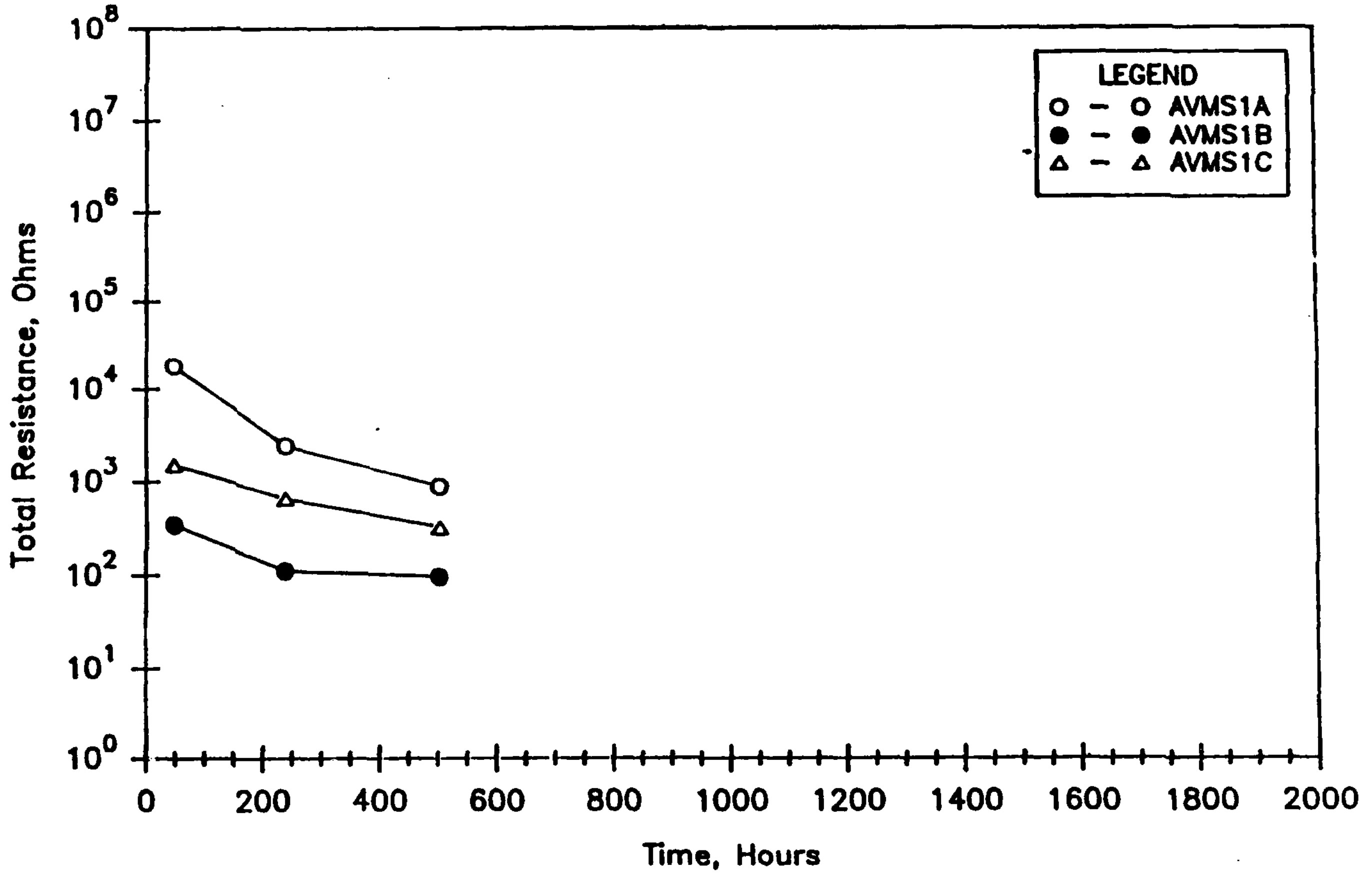


Fig. A-9. Total Resistance as a Function of Time for VMS Coating 2. A and C were Corner Compartments, B was the Center Compartment.



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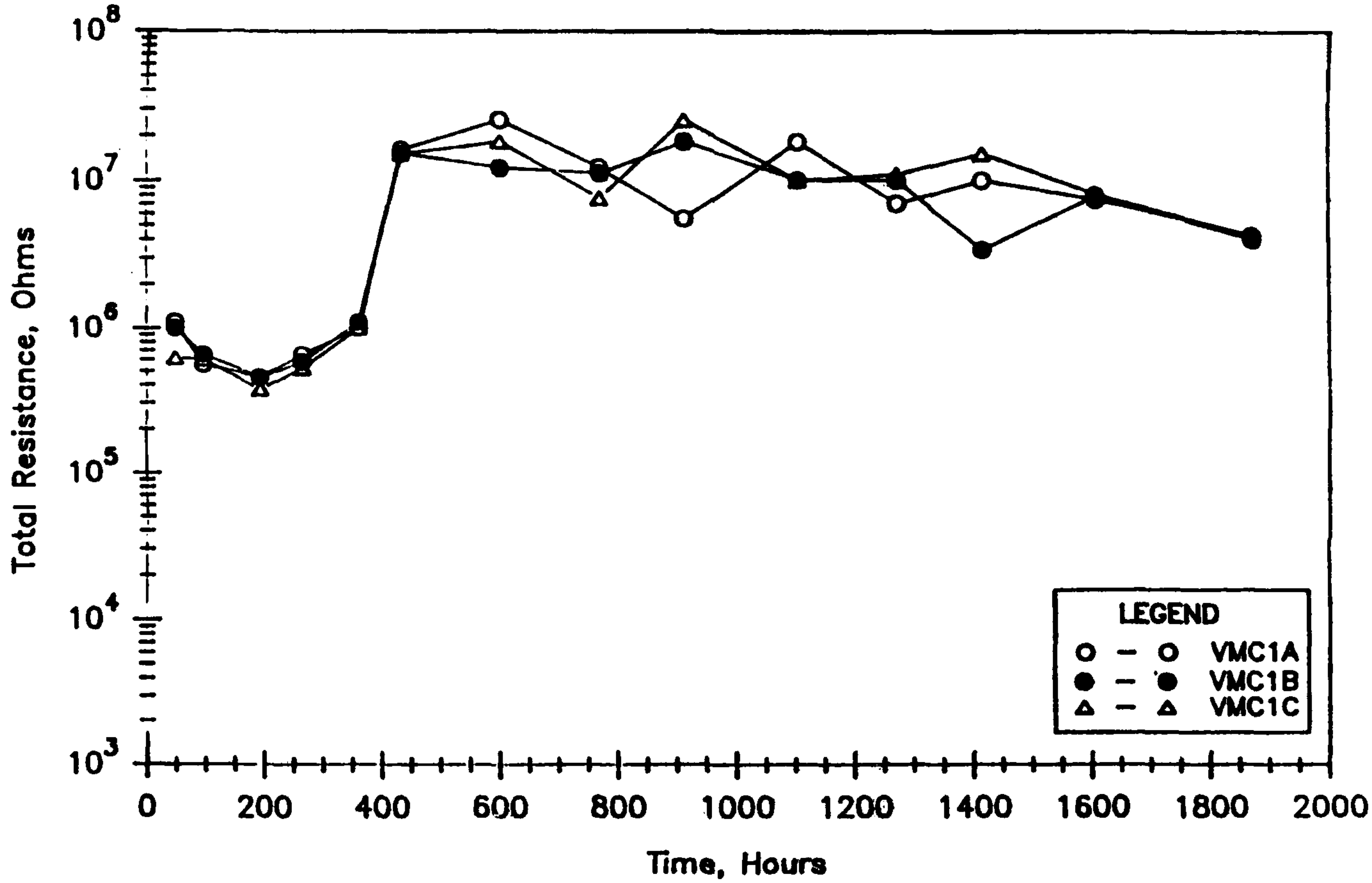

Fig. A-11. Total Resistance as a Function of Time for VMC Coating 1. A and C were Corner Compartments, B was the Center Compartment.

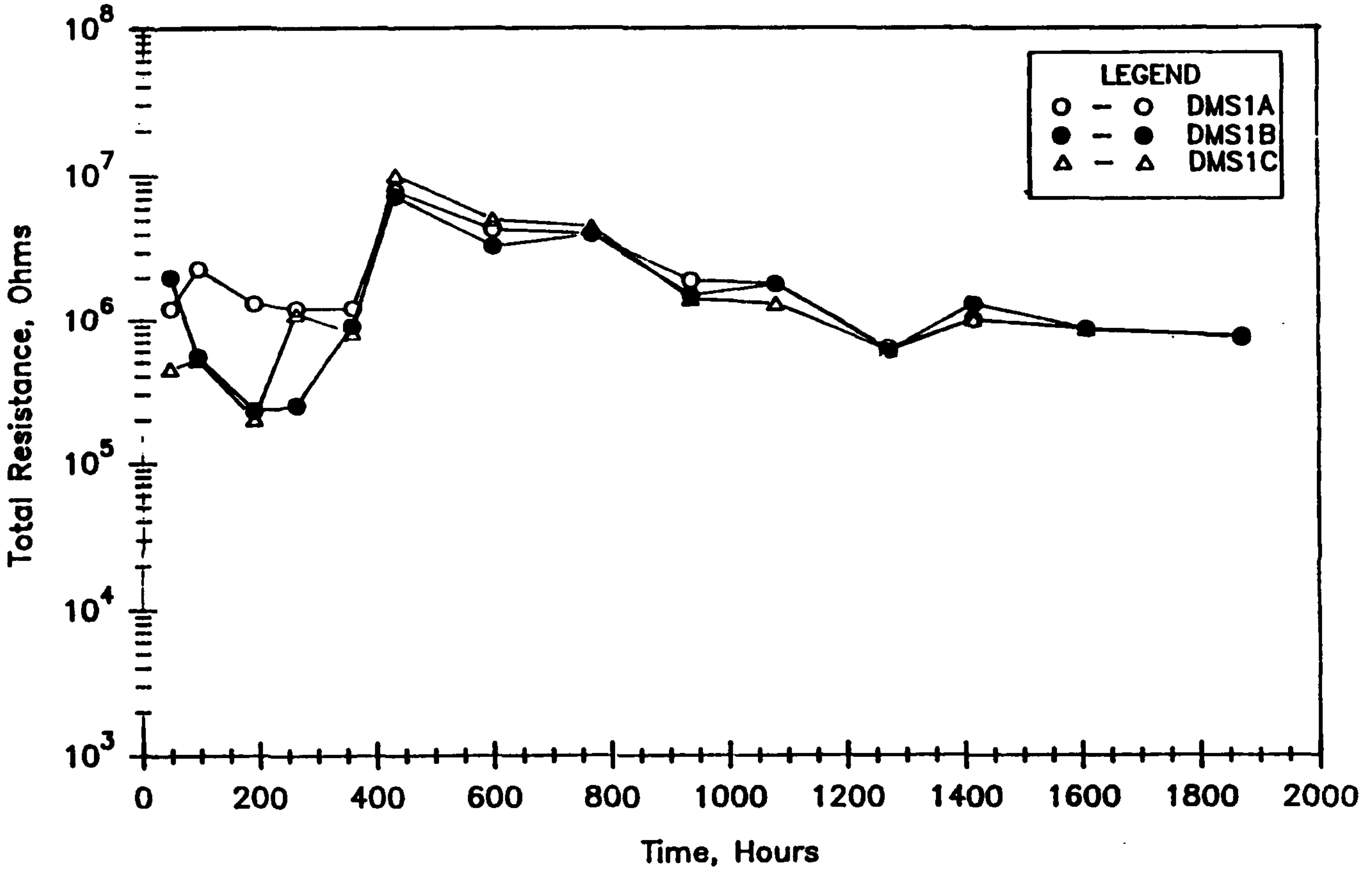

Fig. A-12. Total Resistance as a Function of Time for DMS Coating 1. A and C were Corner Compartments, B was the Center Compartment.

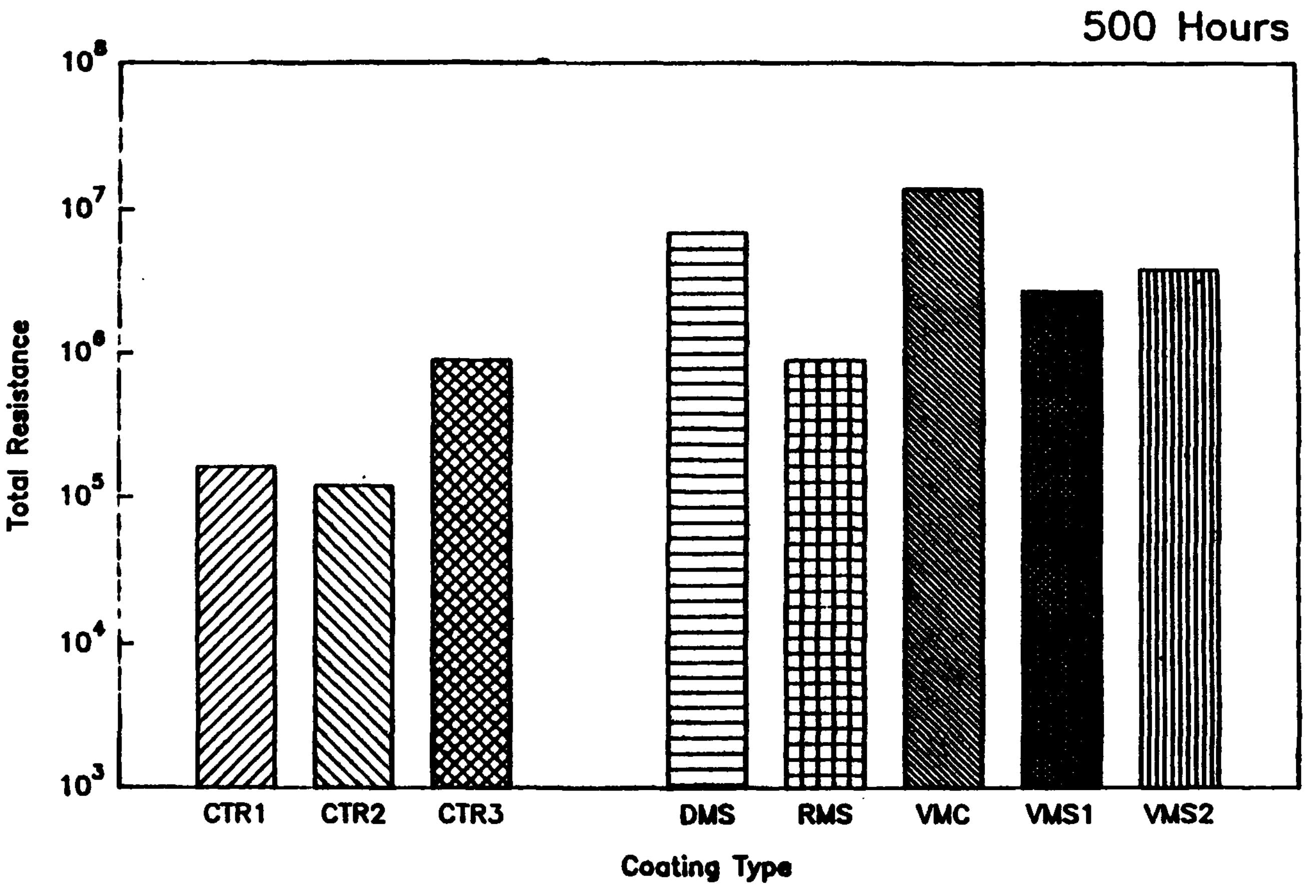

Fig. A-13. Total Resistance for Various Coatings after 500 Hours of Exposure.

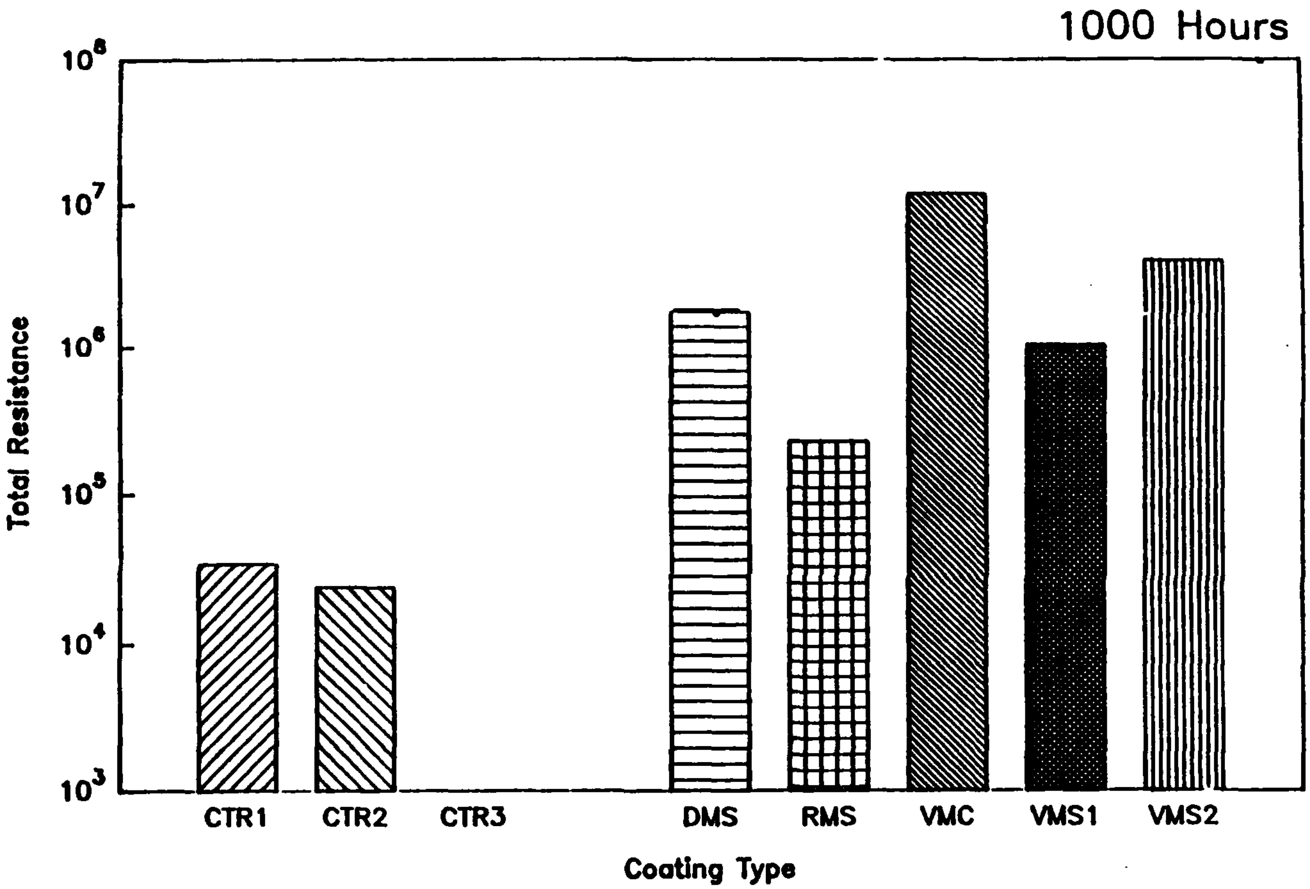

Fig. A-14. Total Resistance for Various Coatings after 1000 Hours of Exposure.

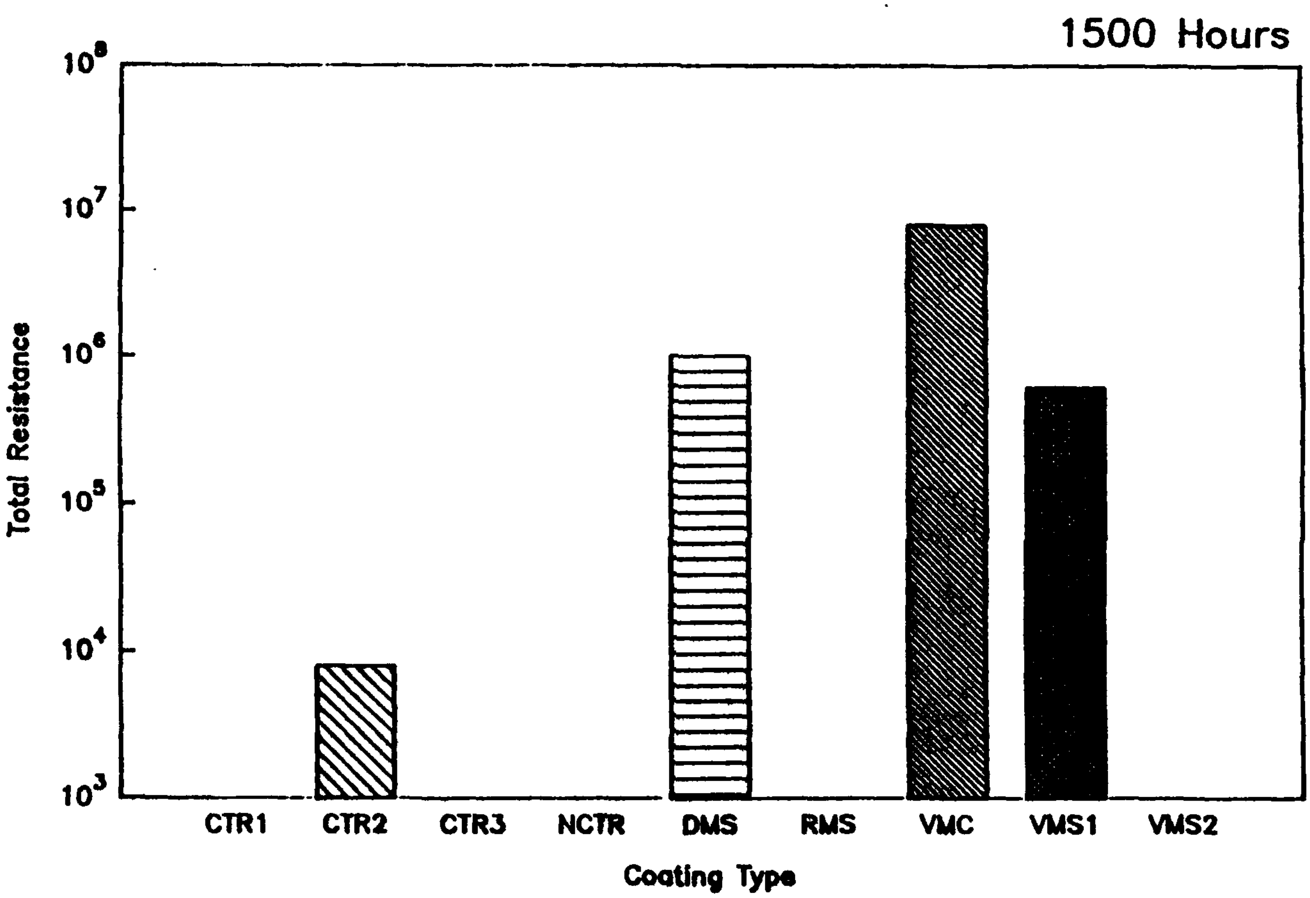

Fig. A-15. Total Resistance for Various Coatings after 1500 Hours of Exposure.

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